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bipyridyl in the title or abstract AND 1945:1974 as the publication date AND ICI as the applicant

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PATENT SPECIFICATION

1035.171



NO DRAWINGS

1035.171

Inventor: RONALD FREDERICK HOMER

Date of filing Complete Specification: April 10, 1963.

Application Date: April 25, 1962.

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Int. Cl.:—C 07 d 31/24 // A 01 n

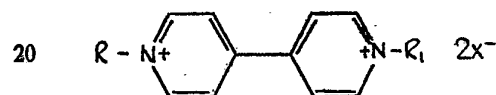
COMPLETE SPECIFICATION

4,4'-Bipyridylum Quaternary Salts

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to herbicidal 4,4'-bipyridylum quaternary salts, to processes for their preparation and to their use as herbicides.

The invention comprises herbicidal 4,4'-bipyridylum quaternary salts having an aliphatic hydrocarbon radical as substituent on each of the nitrogen atoms of the bipyridyl nucleus, one of the said aliphatic radicals being an unsaturated radical and the second being an unsaturated or a saturated radical, and having the formula



where R is an alkenyl radical and R₁ is an alkyl or alkenyl radical and X[−] is an anion.

As unsaturated radical there can be used, for instance, one having 2—8 carbon atoms and especially one having 2, 3 or 4 carbon atoms. Where the groups R and R₁ are both unsaturated aliphatic radicals, these radicals can be the same or different. The saturated radical can be an alkyl radical having 1—8 carbon atoms, and especially a methyl, ethyl, propyl or butyl radical.

The anion X[−] can be, for example, a chloride, bromide or methosulphate ion. The choice of anion depends to a large extent upon the solubility of the respective salts in water and upon the ease with which the salts can be obtained by quaternising 4,4'-dipyridyl

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or one of its mono-quaternary salts. On the whole, it is convenient for the anion to be a chloride ion, in which instance the salts can be obtained using the appropriate chloro-compounds as quaternising agents.

The invention also consists in a process for the preparation of the compounds of the invention, in which 4,4'-bipyridyl or a mono-quaternary salt thereof is quaternised with a suitable quaternising agent. Where a mono-quaternary salt of 4,4'-bipyridyl is used, for instance in the preparation of an unsymmetrical di-quaternary salt, the salt should be so chosen that the substituent on its quaternary nitrogen atom is one of the desired aliphatic radicals in the diquaternary salt, and the quaternising agent used is such that it introduces the required second aliphatic radical. The process can be performed simply by heating a mixture of 4,4'-bipyridyl (or the mono-quaternary salt as the case may be) with the quaternising agent. However, in some instances, especially where operating on a large scale, it may be more convenient to use a suitable liquid as solvent or diluent for the reactants.

The compounds of this invention show herbicidal activity together with the valuable property of being de-activated when they come into contact with soil. Thus the invention also includes herbicidal compositions containing as active ingredient a compound of the invention, and a carrier for the active ingredient. The composition can, for example, be a liquid composition obtained by dissolving or dispersing the active ingredient in a suitable liquid diluent, for example water or a suitable organic liquid. The herbicidal composition alternatively can be in the solid state, for instance a powder or granules, in which the carrier is a suitable solid. Substances which should preferably be avoided for use as the carrier are substances which are highly adsorptive and those which act as cation-exchange materials. The compositions can contain a wetting or dispersing agent

Price 75p

PATENT SPECIFICATION

996,570

NO DRAWINGS.

Inventor:—RALPH SANTORRE FANSHAWE.

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Index at Acceptance:—C2 C(B4A2, B4J).

Int. Cl.:—C 07 d.

COMPLETE SPECIFICATION.

Purification of Bipyridyls.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a process for the purification of organic bases, and more particularly for the purification of bipyridyls.

Bipyridyls are useful intermediates for the manufacture of herbicidal materials, and are commonly made from pyridine by reactions involving oxidation or dehydrogenation. The products of such reactions are usually found to be mixtures made up of various isomeric bipyridyls, the relative proportions of which depend upon the particular reaction conditions employed, in admixture with varying proportions of higher polypyridyls and tarry and highly coloured by-products.

Separation of such products is usually carried out by physical methods, for example fractional distillation, but this is a slow, expensive and inefficient operation on account of the relatively high boiling points of the bipyridyls and the close chemical similarity of the components of the mixtures.

We have now found that there is an unexpected difference in the behaviour of the components of this mixture in a system consisting of water and a substantially water-immiscible organic solvent, and that this difference can be used very effectively for purifying bipyridyls and the close chemical similarity find, dissolve preferentially in the organic solvent phase, but the highly coloured tarry materials and by-products are preferentially dissolved in the aqueous phase. This observation is very surprising, for tarry materials

and by-products are commonly found to be much more soluble in organic solvents than in water.

Thus according to our invention we provide, in a process for the purification of bipyridyls, the steps of contacting an impure bipyridyl with water and a substantially water-immiscible organic solvent and thereafter separating the organic solvent phase, which contains the bipyridyl.

The contacting step is preferably carried out so as to promote intimate contact of the various phases of the mixture and to bring the system rapidly towards the equilibrium compositions for the phases. Conventional means for liquid/liquid extraction may be used for this purpose, for example thorough agitation, which may be produced by mixers, stirrers, agitators or any other convenient device. The separation step may also be carried out by any convenient conventional technique, for example by settling, decantation, centrifugal separators, or the like.

In a simple embodiment of the invention, the impure bipyridyls are mixed with water and the organic solvent and the mixture is agitated, preferably until the system reaches substantially complete equilibrium between the phases, and then the organic solvent phase is separated. The aqueous phase contains most of the tarry and highly coloured impurities and can be discarded, while the organic solvent phase contains most of the bipyridyls. There may be some interference with the separation of the liquid phases owing to the presence of solid phases, though this can be reduced very considerably by carrying out the operation at a temperature above the melting point of any solid organic phase which may be present in the mixture, or by filtering the mixture. In order to re-

[P. 1]

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PATENT SPECIFICATION

NO DRAWINGS

Inventors: RALPH SANTORRE FANSHAWE and CHARLES SHEPHERD

996,569



996,569

Date of filing Complete Specification Feb. 18, 1963.

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Index at acceptance: —C2 C(B4A2, B4J, B4K, B4M)

Int. Cl.: —C 07 d

COMPLETE SPECIFICATION

Separation of 4:4'-bipyridyl from Mixtures Containing it

- We IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- This invention relates to a process for separating 4:4'-bipyridyl from mixtures containing it, and particularly from mixtures of bases produced in processes for making bipyridyls from pyridine.
- 4:4'-bipyridyl is a useful intermediate for the manufacture of herbicidal materials, but corresponding products derived from the various isomeric bipyridyls may differ considerably in their herbicidal activity. Most of the methods at present available for producing bipyridyls give rise to mixtures of isomers, often in admixture with higher polypyridyls, polymeric organic bases and tars. In particular, the 4:4'-bipyridyl produced by oxidation of a sodium-pyridine interaction product contains an appreciable proportion of 2:4'-bipyridyl. As it is commercially desirable to produce herbicidal products in a form having a high concentration of active agent and a standard activity, some inexpensive method for purifying 4:4'-bipyridyl is needed. The separation of 4:4'-bipyridyl from such mixtures by conventional means is not at all easy, especially when the process is carried out on a large scale, because of the similar physical properties of the isomers. The boiling points of 2:4' and 4:4'-bipyridyls are both close to 300°C, for example, and attempts to separate them by fractional distillation are hindered by the tendency of the isomers to co-distil.
- We have now found that 4:4'-bipyridyl can be separated very easily and simply from mixtures containing it, and particularly from admixture with its isomers and other bases, as its hydrate. This hydrate of 4:4'-bipyridyl is substantially a dihydrate, and is relatively insoluble in water.
- Thus according to the present invention we provide an improved process for the separation of 4:4'-bipyridyl from a mixture of isomeric bipyridyls containing it, which comprises treating the said mixture with water and thereafter separating the 4:4'-bipyridyl hydrate at a temperature below about 60°C.
- The process of our invention is especially applicable to the separation of 4:4'-bipyridyl from associated 2:4'-bipyridyl and 2:2'-bipyridyl, and can be used to separate 4:4'-bipyridyl in substantially pure form. Mixtures containing 4:4'-bipyridyl which are particularly suitable for treatment by the process of the present invention include those mixtures of bases, predominantly bipyridyls, produced from metal-pyridine interaction products, for example by oxidation. The mixtures of bases produced from sodium-pyridine interaction products in particular tend to contain much of the 2:4'-isomer in addition to the 4:4'-isomer, and are especially advantageously treated by the present process. The crude products or mixtures of bases produced from magnesium-pyridine and aluminium-pyridine interaction products tend to contain a much higher content of the 4:4'-isomer, but even so our process offers a very simple and rapid method for isolating and purifying the product.
- The mixture containing 4:4'-bipyridyl may be treated directly with water at a temperature below the decomposition temperature of the hydrate (i.e. below approximately 60°C. To minimise the inclusion of impurities within the solid hydrate, however, it is preferred that the mixture containing 4:4'-bipyridyl is treated with water at a tem-



PATENT SPECIFICATION

NO DRAWINGS

999,585

Inventors: JOHN EDWARD DOWNES and DONALD WILLIAM RONALD HEADFORD

Date of filing Complete Specification (under Section 3 (3) of the Patents Act 1949) Jan. 9, 1963.

Application Date: Jan. 22, 1962.

No. 2210/62.

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A5 E(1C3B3, 1C4B3, 1C4B4)

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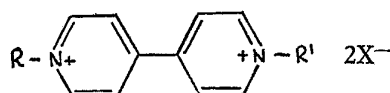
COMPLETE SPECIFICATION

Bipyridylium Compounds and Herbicidal Compositions containing them

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company of Imperial Chemical House, Millbank, London, S.W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

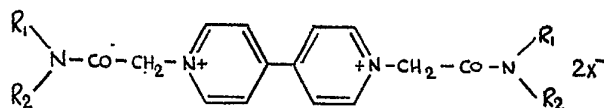
This invention relates to new bipyridylium quaternary salts, to processes for their preparation and to herbicidal compositions containing them.

U.K. Specification No. 813,532 discloses and claims the use as herbicides of compounds of the formula:—



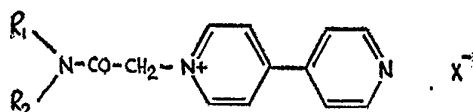
where R and R' are alkyl radicals of not more than four carbon atoms which are substituted with a carbamyl or N-substituted carbamyl group; and X[−] is an anion.

We have now found, according to the present invention, that certain N-substituted carbamyl compounds of the formula shown above show selective herbicidal activity against broad-leaved species compared with their activity against grass species. The selective compounds are those of the formula:—



wherein the two radicals attached to the nitrogen atoms in the bipyridyl nucleus are identical and where R₁ and R₂ together with the nitrogen atom to which they are attached constitute a pyrrolidine, piperidine, hexamethyleneimine or morpholine ring optionally having a methyl radical as substituent; or where R₁ and R₂ are each separate, identical radicals and are alkoxyalkyl radicals of 2, 3 or 4 carbon atoms or cyanoalkyl radicals of 2 or 3 carbon atoms; and X[−] is an anion. The anion X[−] can be a monovalent ion, for example chloride, iodide, or bromide, or a divalent ion — where of course the symbol 2X[−] in the above structural formula represents a single ion rather than two monovalent ions. The choice of anion depends to a large extent upon the solubility of the respective salts in water and upon the ease with which the salts can be prepared.

The invention also consists in a process for the preparation of the compounds of the invention, in which 4,4'-bipyridyl or a mono-quaternary salt of the formula:—





PATENT SPECIFICATION

NO DRAWINGS

1040.170

Inventor: JOHN THEODORE BRAUNHOLTZ

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Application Date: Dec. 20, 1961.

No. 313/66.

(Divided out of No. 1,037,641.)

Complete Specification Published: Aug. 24, 1966.

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Int. Cl.:—C 07 d 31/24 // A 01 n

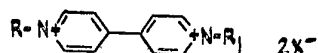
COMPLETE SPECIFICATION

4,4'-Bipyridylum Quaternary Salts

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British company of Imperial Chemical House, Millbank, London, S.W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to 4,4'-bipyridylum quaternary salts, to processes for their preparation and to their use as herbicides.

The invention consists in one aspect of 4,4'-bipyridylum quaternary salts having attached to each of the nitrogen atoms of the 4,4'-bipyridyl nucleus a substituted alkyl radical the substituted alkyl radicals being dissimilar. The bipyridylum quaternary salts can be, for example, compounds of the formula:—



where R and R₁ are dissimilar and are alkyl radicals of 1—4 carbon atoms bearing substituents, and X[−] is an anion.

The substituents in the alkyl radicals referred to above can be, for instance, chosen from the following:—hydroxyl, halogen, carbonyl, alkoxy, carboalkoxy, carbamyl N-substituted carbamoyl, amino, substituted amino, phenyl, substituted phenyl, acyl and substituted benzoyl. Thus, R₁ in the formula shown immediately above can be an ethoxycarbonylalkyl, hydroxyalkyl, acetyl or carbamoylalkyl radical.

Compounds which have been found to possess good herbicidal activity are the following:—

1 - (β - hydroxyethyl) - 1' - acetyl - 4,4'-bipyridylum quaternary salts; and

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1 - (N,N - dialkylcarbamoylmethyl) - 1' - (N,N - dialkylcarbamoylmethyl) - 4,4'-bipyridylum diquaternary salts in which the two N,N-dialkylcarbamoylmethyl groups are different from each other.

The anion of the compounds of the invention can be a monovalent ion, for example a chloride, bromide or methosulphate ion, or a divalent ion in which instance, of course, the symbol 2X[−] in the above structural formulae represents a single ion rather than two monovalent ions. The choice of anion depends to a large extent upon the solubility of the respective salts in water and upon the ease with which the salts can be obtained.

The compounds of the invention show herbicidal activity together with the valuable property of being deactivated when they come into contact with soil. Thus the invention also consists in herbicidal compositions comprising as active ingredient a 4,4'-bipyridylum quaternary salt of the invention and a suitable diluent for the active ingredient. The compositions can be, for example, liquid compositions in which the active ingredient is in solution or dispersion in water or a suitable organic solvent. The compositions can alternatively be powder compositions in which the diluent is a suitable finely-divided substance, for instance china clay or talc. The ingredients used with the active ingredient in the herbicidal compositions of this invention can be substances known to the art as being suitable in the formulation of herbicidal compositions, for instance surface active substances such as wetting and dispersing agents, binders, stickers, corrosion inhibitors, stabilising agents and colouring agents.

Any wetting or dispersing agent used in the herbicidal compositions should preferably be a non-ionic surface active compound, for instance an ethylene oxide—nonyl phenol condensate or a cationic compound, so as to avoid any un-



PATENT SPECIFICATION

NO DRAWINGS

963,441

Inventors: ALBERT BAINES and ALASTAIR CAMPBELL*Date of filing Complete Specification:* May 10, 1962.*Application Date:* May 26, 1961.*No.* 19081/61.*Complete Specification Published:* July 8, 1964.

© Crown Copyright 1964.

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International Classification:—C 07 d

COMPLETE SPECIFICATION

Process for the production of Bipyridyls

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an organic chemical process for the manufacture of bipyridyls, more particularly for the manufacture of 4:4'-bipyridyl.

It is known that bipyridyls, including particularly 4:4'-bipyridyl, can be prepared by oxidation of the product formed by the interaction of sodium and pyridine. Manufacture of bipyridyls in this way is complicated by the difficulty encountered in making the interaction of sodium and pyridine proceed in a rapid but controllable manner, and by the risk of explosion on the oxidation stage consequent on the presence of unreacted sodium. It has been proposed to carry out the sodium-pyridine interaction in the cold, to use a very large excess of pyridine, or to heat the pyridine-sodium interaction product for long periods in order to achieve satisfactory regulation of the reaction or to improve the yields of bipyridyls. These proposals are not economically attractive or convenient, however, and the yields of bipyridyls so obtained are not high.

The prior art processes also have the disadvantage that the small yield of bipyridyl isomers thereby produced contain considerable amounts of the 2:2'- and 2:4'-isomers in addition to the 4:4'-isomer. The 4:4'-isomer is a particularly valuable intermediate for the manufacture of herbicidal materials, and any formation of the other isomers consequently represents a loss of expensive starting materials. It is therefore of importance that the yield of bipyridyls and the proportion of the 4:4'-isomer in the product should be increased as much as possible.

We have now found that the ratio of 4:4'-isomer to the other bipyridyl isomers obtained by the oxidation of the product formed by the interaction of sodium and pyridine varies with the conditions, particularly the temperature of the initial reaction between the sodium and the pyridine. We have also found that the separation of the products of the oxidation step (which is complicated by the formation of a dispersion of sodium hydroxide in a colloidal and unfilterable form) can be simplified by adding water to the oxidised reaction mixture, and heating for a short time.

According to the present invention, therefore, we provide a process for the manufacture of bipyridyls which comprises treating a pyridine with sodium at a temperature of at least 50°C. and oxidising, as hereinafter defined, the sodium-pyridine interaction product so formed.

The process is carried out in the liquid phase, and we find that especially in the case of pyridine itself the most suitable temperature for treatment of the pyridine with the sodium is in the range 50°C. to 120°C., and preferably in the range 75°C. to 100°C.

The examples illustrating the invention show clearly that the amount of the unwanted 2:4'- and 2:2'-isomers falls as the temperature of the reaction between sodium and pyridine is reduced from 115°C. to 80°C. At 115°C. the ratio of the amount of 4:4'-isomer to the amount of the other isomers in the final product is of the order of 1.6 to 1, and at 90°C. this ratio is of the order 14 to 1. It is important to note that if the temperature is reduced to below 50°C. the yield on both sodium and pyridine falls and an increased amount of undesirable by-products is produced.

It is clear therefore that it is of importance to control the temperature of the reaction between the sodium and the pyridine in order to obtain a high proportion of 4:4'-bipyridyl

[Pric 441]

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2,962,502

PREPARATION OF 2,2'-BIPYRIDYL

Peter Frank Hilary Freeman and Ranajit Ghosh, Bracknell, England, assigns to Imperial Chemical Industries Limited, London, England, a corporation of Great Britain

No Drawing. Filed Jan. 5, 1959, Ser. No. 784,879

Claims priority, application Great Britain Jan. 24, 1958

5 Claims. (Cl. 260—296)

This invention relates to a new manufacturing process and more particularly it relates to a new process for the manufacture of 2:2'-bipyridyl from pyridine.

It has been proposed to manufacture 2:2'-bipyridyl from pyridine by oxidation thereof with ferric chloride. This known process is unsatisfactory in that the yield of 2:2'-bipyridyl is low. Numerous side-reactions take place leading to by-products.

We have now found that these disadvantages may be obviated by oxidising pyridine by means of ferrous chloride. It is surprising that ferrous chloride can be used in this manner since it does not normally act as an oxidising agent.

According to the invention we thus provide a process for the manufacture of 2:2'-bipyridyl which comprises heating pyridine with ferrous chloride. The process of the invention may conveniently be carried out at superatmospheric pressure for example in a sealed tube, or an autoclave or other pressure vessel.

The process is preferably carried out at temperatures between 250° C. and 350° C., preferably between 270° C. and 340° C. At temperatures below 250° C. the reaction is inconveniently slow and at temperatures above 350° C. considerable decomposition takes place. Nevertheless in the operation of a continuous process or in other instances where a short reaction time is desired, possibly the use of temperatures in the region of 350° C. may be advantageous.

The proportions of pyridine and ferrous chloride used in the process of the invention may be varied within wide limits. The process may for example be successfully carried out with molar ratios of pyridine to ferrous chloride between 3 to 1 and 25 to 1.

The ferrous chloride used in the process of the invention may be in the hydrated form or it may be anhydrous and it may be added to the pyridine as free ferrous chloride or as the hydrate or again in the form of a ferrous chloride-pyridine complex.

The 2:2'-bipyridyl may be isolated from the reaction mixture by any convenient means known to the art, for example by separation therefrom as an iron complex, by

2

steam distillation, by distillation or solvent extraction. The excess of pyridine may be recovered and again used for the process.

2:2'-bipyridyl is a valuable intermediate compound for use for example in the manufacture of the herbicidal compounds for example 1:1'-ethylene-2:2'-dipyridylium dibromide.

The invention is illustrated but not limited by the following example in which the parts are by weight:

Example

A mixture of 40 parts of ferrous chloride tetrahydrate and 78 parts of pyridine is heated in a sealed tube during 20 hours. The contents of the tube are then cooled and mixed with 2,500 parts of water. The mixture is made strongly alkaline by adding a solution of 600 parts of sodium hydroxide in 800 parts of water and it is then subjected to steam distillation. To the distillate there is added aqueous hydrochloric acid until the pH value is 1.0. The mixture is then evaporated to dryness and the residue is made strongly alkaline by the addition of sodium hydroxide solution. It is then extracted with ether and the extract is fractionally distilled. There is obtained 60.4 parts of unchanged pyridine and 13 parts of 2:2'-bipyridyl M.P. 68° C. to 69° C. This corresponds to a yield of 75% based on the pyridine consumed.

What we claim is:

1. Process for the manufacture of 2:2'-bipyridyl which comprises heating pyridine with ferrous chloride at superatmospheric pressure and a temperature between about 250° C. and 350° C., whereby said pyridine is oxidized to 2:2'-dipyridyl, the ferrous chloride constituting the sole essential oxidizing agent.

2. Process as claimed in claim 1 wherein the reaction temperature is between 270° C. and 340° C.

3. Process as claimed in claim 1 wherein the molar ratio of pyridine to ferrous chloride is between 3 to 1 and 25 to 1.

4. Process as claimed in claim 1 wherein the ferrous chloride is in the form selected from the group consisting of the anhydrous salt, the hydrate and ferrous chloride-pyridine complex.

5. Process for the manufacture of 2:2'-bipyridyl which comprises heating pyridine with ferrous chloride at a temperature between about 250° C. and 350° C. and superatmospheric pressure using a molar ratio of pyridine to ferrous chloride between 3 to 1 and 25 to 1, and thereafter isolating the resulting 2:2'-dipyridyl from the reaction mixture.

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Hein et al.: Ber. Deut. Chem., vol. 61B, pp. 1790-1791 (1928).

(19)		Canadian Intellectual Property Office	Office de la Propriété Intellectuelle du Canada	(11)	CA 783065	(13)	A
		An Agency of Industry Canada	Un organisme d'industrie Canada	(40)	16.04.1968		

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(21) Application number: **783065D**

(51) Int. Cl:

(22) Date of filing: ..

(71) Applicant: **ICI LTD.**

(72) Inventor: **OLLEVEANT ALLAN W ().
FANSHAWE RALPH S ().**

(54) **BIPYRIDYL PRODUCTION**

(57) **Abstract:**

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(12)

(21) Application number: **784730D**

(51) Int. Cl:

(22) Date of filing: ..

(71) Applicant: **ICI LTD.**

(72) Inventor: **CAMPBELL ALASTAIR ().
BRADBURY FRANK R ().**

(54) **BIPYRIDYL PRODUCTION**

(57) **Abstract:**

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(12)

(21) Application number: **882721D**

(51) Int. Cl:

(22) Date of filing: ..

(71) Applicant: **ICI LTD.**

(72) Inventor: **WADDAN DHAFIR Y ().
WILLIAMS DEREK ().**

(54) **PREPARATION OF 2,2'-BIPYRIDYL**

(57) **Abstract:**

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